The Commercial Stainless Steel Tube Enveloping Technique for MgB_2

H. B. Lee, B. J. Kim, Y. C. Kim[a)]

Department of Physics, Pusan National University, Busan 609-735, Korea

D. Y. Jeong

Korea Electrotechnology Research Institute, Changwon 641-120, Korea

Abstract

A commercial stainless steel tube was employed to fabricate MgB_2. The specimen was prepared by a stoichiometric mixture of Mg and B inside the stainless steel tube. The specimen was sintered for 2 hours at 920^{o}C. X-ray spectra showed there were no second phases like MgO. The transition temperature of the specimen was 37.5 K with a sharp transition width within 1K. The specimen showed a good connection between grains and critical current density as calculated with the Bean model is more than 10^5 A/cm^2 in the 20 K and zero field.

PACS numbers : 74.60.-w; 74.70.Ad; 89.20.Bb

a) Corresponding author: Tel 82 - 51 - 510 - 2224, e-mail : yckim@pusan.ac.kr

After Akimisu announced the discovery of 39K-temperature superconductivity in MgB_2, it has stimulated considerable interests in superconducting research groups.[1] In the early study, it was reported that MgB_2 superconducting phase was fabricated under the condition of a high temperature and high pressure.[2] But it was soon revealed that high pressure was needed to prevent Mg from escaping from the base materials.[3, 4] It is because Mg is a too volatile material. Many groups reported that if Mg did not escape from the base materials in the elevated temperature, MgB_2 was fabricated.[5, 6]

An effective way to prevent the escape of some kinds of elements in the base materials during heat treatment is envelope treating. It has been mainly used when low-melting-point and high-evaporating materials are heat treated, especially Tl, Hg, Mg, etc.[4, 7, 8] From now on, refractory metals and quartz tube are mainly used for materials of enveloping.[3, 4] Ta and Nb are candidate of refractory metals. These materials have a high melting point and a strong Mg corrosion resistance.[9] But in the elevated temperature, these materials are like to oxidize. Because of the high reactivity of Mg with oxygen, even a small hole can make it very bad to fabricate MgB_2. To prevent the surface from oxidizing, we have to treat specimens in inert gas or in a vacuum state. This is another problem of rising cost, especially fast-heat and fast-cool treating. Recently many groups used to envelope a quartz tube to outside Ta envelope once more and evacuate quartz tube to avoid surface oxidation of Ta.[3, 6]

The quartz tube for the envelope treating was used for a rather lower fabricating temperature than for MgB_2, like Hg-based and Tl-based superconductors.[4, 7, 8] Use of the quartz tube for fabricating the MgB_2 superconductor was restrained owing to softening of the quartz tube in the elevated temperature(around 900°C) and the reaction of Si with Mg. So the quartz tube cannot be used but rather a second material to avoid surface oxidation in fabricating the MgB_2 superconcuctor.

In the case of short films with the MgB_2 superconductor, the refractory metals and the quartz tube for the envelope are believable, but rather inconvenient for Mg's affinity for a oxygen and high cost. With respect to fabricating long-length wires and films with the MgB_2 superconductor, these are more difficult to fabricate owing to the possibility of breakage in using the quartz tube and the cost of fabricatation.

So, there are deep demands that are an economical and easy treatment method for envelope technology. Now, we have developed the Commercial Stainless Steel Tube Envelope

Technique(COSSET) for the MgB_2 superconductor. The COSSET is easy, economical and believable. The COSSET is hard enough to be in a longer heat treatment of the elevated temperature and is not breakable like the quartz tube and does not need special environments like Ta and Nb. It will be very useful for long length wires and films of MgB_2.

The starting materials are Mg(99.9% powder)and B(96.6% amorphous powder). The sample of MgB_2 was prepared in several steps. Mixed Mg and B stoichiometry was finely ground, then pressed into a pellet 10mm in diameter. Also, an 8m-long stainless steel(304) tube was cut into a 10cm piece. One side of the 10cm long tube forged and welded and Fe plate was inserted into the stainless steel tube. The pellet was put on the Fe plate. The pellet had been heat treated at 300°C for 1hr to harden it before insertion into the stainless steel tube. Excess Mg was put under the Fe plate and the other side of the stainless steel tube was forged and put into a high-purity Ar gas in the stainless steel tube, and which was welded. Finally, it was heat treated at 920°C for 2hrs using the fast-heat and fast-cool method in air.[8]

Figure 1 shows the XRD pattern of the MgB_2 bulk sample heat treated at 920°C for 2hrs. There are no second phases like with MgO in the XRD pattern. Second phases like MgO itself do not harm the superconductivity, because it acts as a pinning center. But during the process to fabricate MgB_2, the existence of MgO means that base materials(Mg and B) have reacted with outside oxygen. That would make the superconducting parts small and the non-superconducting parts large, and would drop the confidence of superconductivity. By comparing the COSSET with other processes such as high pressure,[10, 11] PIT(powder in tube)[5] and simple wrapping in iron plate,[12] the COSSET efficiently restrained the oxygen from their source (the outside of the envelope). This would lead us to believe that the sample has good property of MgB_2 in the COSSET.

Figure 2 shows the SEM photographs of the MgB_2 bulk sample. Part(a) shows the surface of the pellet, and part(b) shows the inside of the pellet. Part(b) shows different results from MgB_2 fabricated by other processes such as high pressure, PIT or simple wrapping. The inside of the pellet was condensed by the COSSET during the fabrication of MgB_2. It is not surprising that MgB_2 was condensed, because each Mg and B were composed of one MgB_2. The condensing of the specimen would give rise to a porosity. We have not found a micro structure like this in other reports. And we could also learn that the MgB_2 grains are connected to each other by some kinds of chains. This is another special aspect of the

COSSET.

Generally when one fabricates MgB_2 by another process, an excess Mg is needed. After fabricating MgB_2, the excess Mg can be harmful if not finely dispersed. Unlike other processes, in the COSSET excess Mg was added separately to the MgB_2 pellet and there was no non-dispersed Mg in the MgB_2 superconducting phase. In other words, because the excess Mg is supposed to take part in the reaction by a vapor through the porosity, the non-dispersed excess Mg is almost non-existent in the MgB_2 superconducting phase.

The resistance versus temperature curve and the magnetic susceptibility versus temperature curve of the MgB_2 bulk sample are shown in Fig. 3. It is clear that the superconducting transition temperature in the resistance vs temperature curve at 50mA was about 37.5K and the transition was sharp and the transition width was within 1K, indicating the good quality of the sample. The magnetic susceptibility for the sample was measured at 5 Oe. The magnetic susceptibility date shows that the superconducting transition width was also within 1K. The decreased field cooling signal in the magnetic susceptibility for the sample indicates that the flux pinning was greatly enhanced and suggests a higher possibility of high current superconducting applications in the bulk form and films by the COSSET. The transition temperature of the sample was also about 37.5K.

The M-H curves in Fig. 4 were measured in the temperature region from 5K to 35K. The symmetry in the increasing and the decreasing field branches was good. This means that the contribution of the bulk pinning dominated. On the other hand, the shapes of the M-H curve in the Fig. 4 have a good similarity to each other. One remarkable feature is that flux jumping was shown up to the temperature of 15K. Flux jumping for MgB_2, which refers to a sudden dissipative rearrangement of magnetic flux within a superconductor, was reported by many groups, especially bulk samples.[5, 12, 13] The flux jumps of the sample by the COSSET are rather different from ones of other processes. Most reports show that flux jumps appear within the temperature of 10K. It was well known that impurities and second phases existent in the specimen would influence flux jumps. The impurities and second phases pin the flux and stop the flux jumps. This is also the reason that the films with the MgB_2 superconductor are little affected by flux jumps. In our experiments, there is no proof of second phases, and it is considered that this fact increases the temperature of flux jumps.

The critical current density(Jc) versus applied fields curves of the MgB_2 sample were

obtained from the Bean model(the formula Jc=15ΔM/a(1-a/3b), where 2a is the sample thickness and 2b is the sample width) and are shown in the Fig. 5. Jc reaches more than 1×10^5A/cm^2 at the temperature of 20K in zero field and 3.3×10^4A/cm^2 at 20K in 1T. These values of the sample are as much as or more than ones of other methods in spite of the COSSET's benefits and the high porosity of the sample.

In summary, by using the Commercial Stainless Steel Envelope Technique (COSSET), we could successfully synthesize high-quality MgB_2 for which the critical temperature was 37.5K. The MgB_2 has high-porosity structure and there is no proof of the MgO phase. The magnetic transport J_c of the sample is as much as or more than that of other methods in spite of the COSSET's benefits and the high porosity of the sample. The COSSET will be a good method for the practical application of MgB_2.

Acknowledgments

This work was supported by grant No. R14-2002-029-01000-0 from ABRL Program of the Korea Science and Engineering Foundation.

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FIGURE CAPTIONS

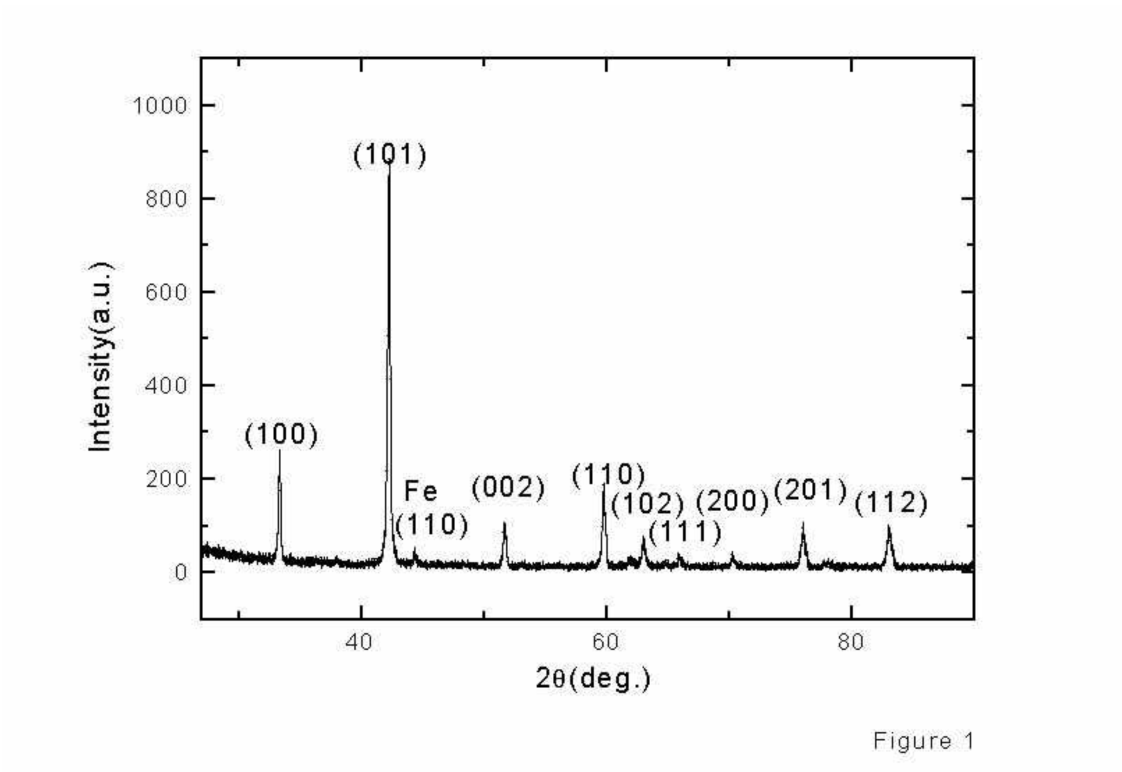
FIG. 1. The XRD pattern of the MgB_2 bulk sample by the COSSET. There is no MgO peak in this pattern.

FIG. 2. The scanning electron micrograph of the MgB_2 bulk sample by the COSSET: (a) on surface and (b) inside.

FIG. 3. The magnetic susceptibility versus temperature curve of the MgB_2 bulk sample by the COSSET. The inset shows resistance versus temperature dependance for the same sample.

FIG. 4. The M-H curves for the MgB_2 bulk sample by the COSSET.

FIG. 5. The critical current density versus magnetic field curves of 5 K $<$ T $<$ 35 K for the MgB_2 bulk sample by the COSSET.



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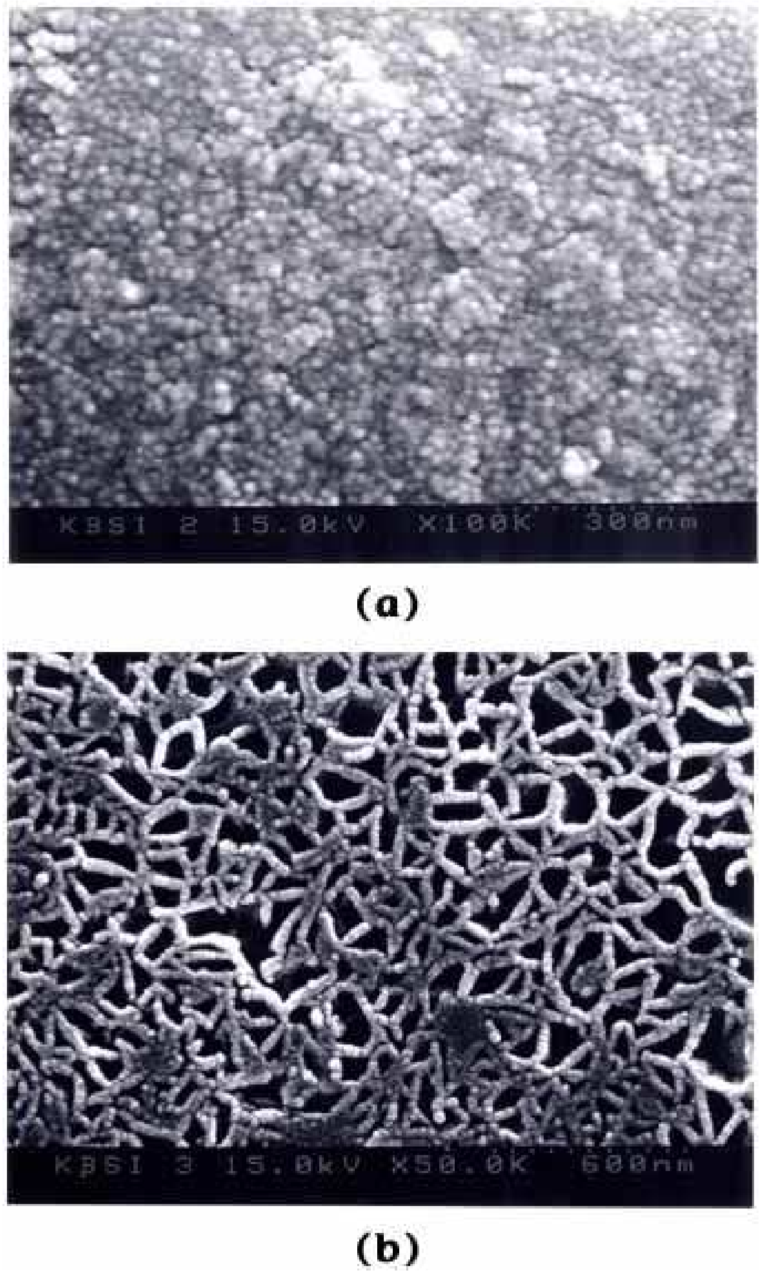

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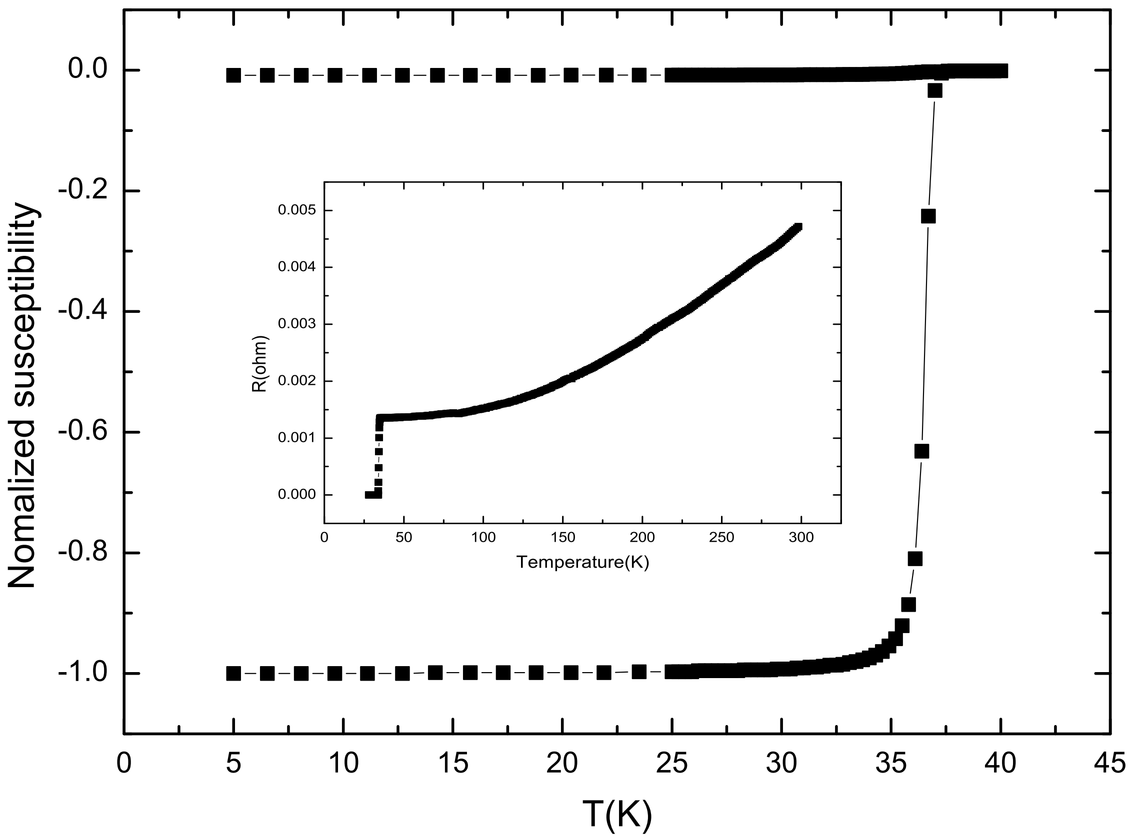

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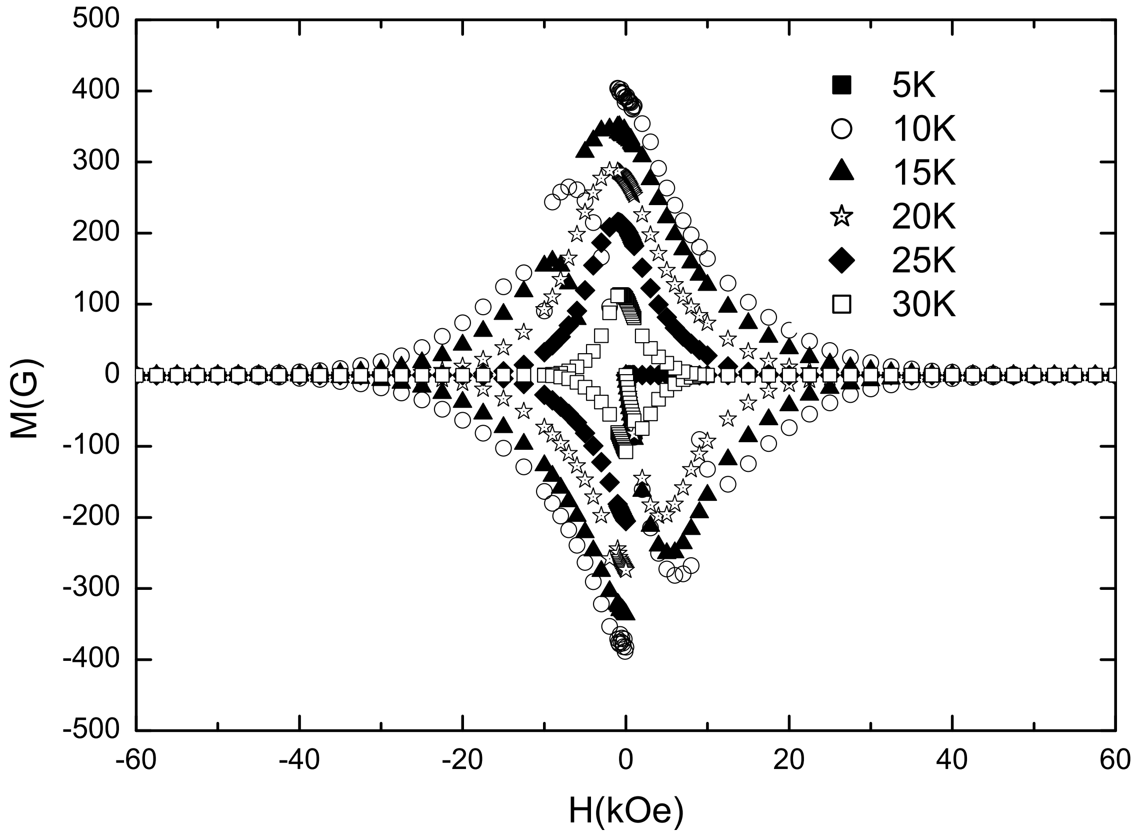

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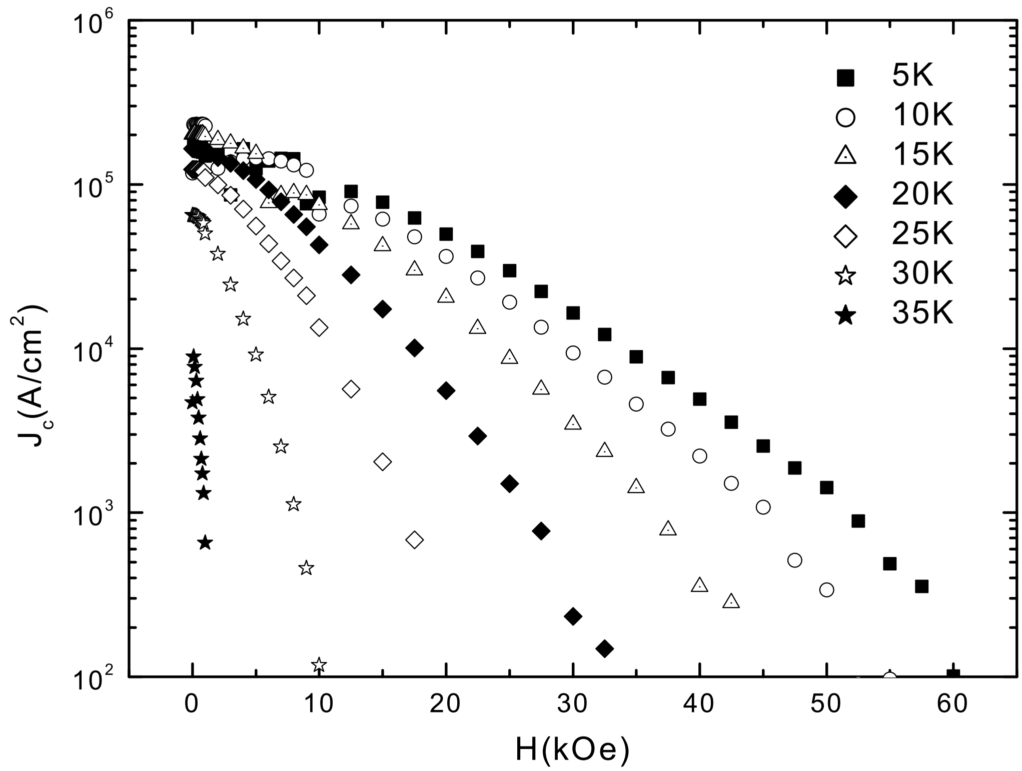

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